

Monitoring Sealant Durability during Instrumented Outdoor Exposure with Variation in Prestrain

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Introduction

Sealants are a vital part of a building's environmental barrier envelope. Their ability to bond to numerous dissimilar substrates and form cure-in-place seals results in their widespread use throughout single story and high rise buildings. A typical 40 story concrete and glass facade building can have 70 miles of sealant bonds. Failure of sealants can lead to energy loss and widespread moisture ingress resulting in cosmetic and structural damage as well as the potential for mold colonization. Therefore, increasing the durability of sealants is of significant economic value. In this research program we are seeking to discern the significance of environmental and strain effects on sealant durability. Sealant bonded specimens are fixtured on automated test instruments outdoors that impose hot compression/cold tension strain cycling in accordance with the temperature of exterior panels simulated here by a section of poly(vinyl chloride) (PVC) pipe. The effect of application conditions is investigated by prestraining the sealants. Herein we report the results of 17 months of continuous outdoor exposure.

Experimental

Specimens: The sealant samples were provided by Dow Corning and an unnamed company referred to here as Company X. Dow Corning provided a one part neutral condensation cure room temperature vulcanizing (RTV) silicone. Company X provided a RTV polyurethane sealant. The adherends consist of anodized 6063 aluminum blocks (76.2×12.7×12.7mm) bonded together with sealant in the form of a 50.8×12.7×12.7mm bond line cured in conformance with ASTM C719.¹

Strain Cycling: Sealant specimens were mounted in two instruments consisting of a fixed upper beam and a moveable lower beam. The instruments are referred to as IIIA, figure 1, and IIIB were custom built at the Forest Products Laboratory.² Strain was imposed on the specimens by moving the lower beam up and down with linear actuators in accordance with a predetermined algorithm. The algorithm simulated actual weatherproofing applications where joint sealants are compressed as the building heats up and expands, referred to as hot compression.

Strain: The instrument displacements were controlled by a computer in accordance with equation 1 where Δ is the displacement in centimeters and T is the measured temperature in degrees Celsius of a section of gray PVC pipe exposed to the weather. Parameters were selected such that the specimens would experience strains between +25% and -25% corresponding to temperatures of between

-18°C and 38°C with zero strain at 10°C.

$$\Delta = (10 - T)/8.75 \quad (1)$$

Additionally, 1/3 of the specimens were mounted so that they were unstrained when the instrument was at its neutral position, 1/3 of the specimens were mounted with +20% prestrain and another 1/3 were mounted with -20% prestrain. These specimens would experience -25 to +25% strain, -5 to +45% strain and -45 to +5% strain over the temperature range, respectively. These prestrains simulated cases where sealants were installed in concrete/steel/polymer constructed building joints that were smaller or larger than their average gap because of the thermal state of the building at the time of sealant installation. The range of -25 - +25% strain was selected as a typical value of strain rating for building sealants.

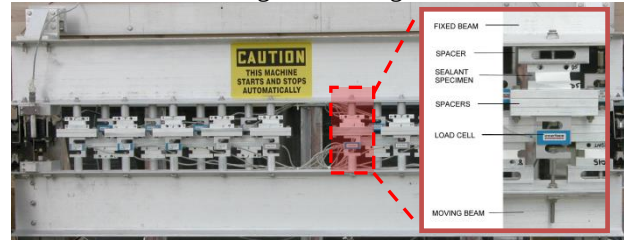


Figure 1: Photograph of IIIA with sealant specimens. The inset image is a close up of one assembly.

Modulus: Twice a week environmental displacement cycling was stopped and an apparent modulus (E_a) cycle was run to check for changes in E_a resulting from weather and cyclic displacement aging. The cycle consisted of a 25 minute zero strain hold followed by compressive and tension segments consisting of two 15% strain peaks that act to remove Mullins effects from the sealants and a 10% strain stress relaxation period. The de-Mullins peaks are intended to remove any effects of filler bonds and secondary bonds that contribute to non-reversible load-displacement behavior.³ Thus, the stress relaxation period occurs at lower strains than the first two peaks and is free of these effects.

E_a is determined using equation 2, where t is time, λ is extension ratio, L is the load, W is initial specimen width, and B is specimen thickness. λ is calculated using equation 3 where h is the initial specimen height. This methodology is taken from the statistical theory of rubber elasticity.³

$$E_a(t, \lambda) = \frac{3L(t)}{WB(\lambda - \lambda^{-2})} \quad (2) \quad \lambda = 1 + \Delta/h \quad (3)$$

Weather: Temperatures were measured using T type (copper-constantan) thermocouples. Both ambient air tem-

perature and the temperature of the PVC pipe were recorded. Relative humidity and solar irradiance was also measured and recorded. Additional weather data was obtained from the National Weather Service as recorded at the Madison Airport located approximately 20 km from the test site. Sealant exposure started in November 2011 and is still continuing. Presented here are the findings for exposure through April, 2014.

Results and Discussion

The daily delta strain variation and average strains imposed to the sealant specimens for instrument IIIa are shown in Figure 2 along with the average PVC drive temperature for the day. Delta strain generally varies within the range of 10-25% during the testing period with broader variations taking place mostly in the spring. Average strain inversely correlates with temperature due to the prescribed hot compression testing mode. During the testing period the average strain ranged from -24 to 30% with average temperatures ranging from 35 to -23°C. The daily average strain reached a value of +55% during January of 2013 after a lightning strike caused a LVDT failure allowing IIIa to run to its maximum tensile limit. All sealants were partially debonded due to this high strain excursion and thus analysis proceeded only with IIIB instrument data as it did not experience LVDT failure. This effectively reduces the replicate count to 2 after this occurrence.

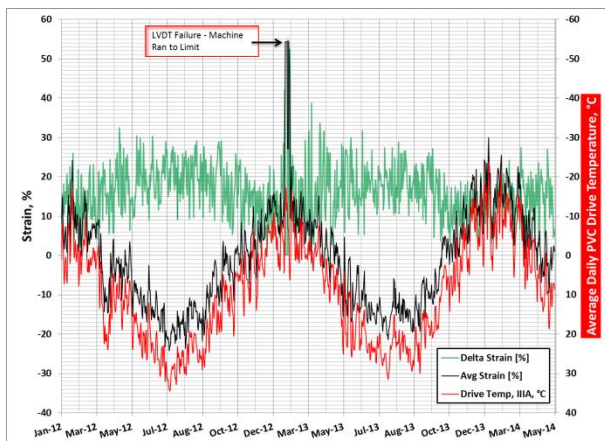


Figure 2: Daily delta and average strain, and average PVC drive temperature for IIIA (scale inverted).

An example of the sealant's stress responses to daily strain cycling is shown in Figure 3 for the urethane, top, and silicone, bottom, specimens during the month of March 2012. The temperature shown in figure 3 is the PVC drive temperature which is strongly affected by radiative heating during the day. March 2012 started with typical winter temperatures with a sharp drop to -13°C on the morning of the 5th, followed by a rapid climb to an unseasonable high of 16°C. This was followed by 24 days of summer like weather with the PVC temperature reaching a high of 41°C on March 15th only to see it fall 34° to a low of 7°C that night. The month ended with temperatures closer to seasonable averages. Figure 3 illustrates the ex-

treme stress variation experienced by the sealants throughout the month of March.

The silicone sealants exhibit significantly higher stress response than the urethane likely due to its higher modulus of elasticity. The absolute stress for both sealants are highest for the specimens with the -20% prestrain followed by the 0, and +20% prestrain specimens. The peaks in daily compressive stress that correspond to daily high temperatures are not individual peaks but can be composed of a multitude of cyclic stresses superimposed on the gradual rise and fall of compressive stress during the day. The inset in the bottom left hand corner of figure 3 highlights the erratic cyclic stresses that occurred on March 17. The inset in the bottom right hand corner of figure 3 shows the stress versus strain behavior for the 20% prestrain silicon specimen. Many overlaid stress loops result from rapid heating and cooling caused by variable cloud cover 17. This rapid cycling occurs when the PVC drive temperature rapidly rises or falls in response to radiative heating fluctuations caused by changes in cloud cover. Overcast winter days such experienced in the beginning and end of the March have very few such peaks. However, hot partly cloudy days experienced in the middle of March have higher mean stress and erratic cyclic behavior. These conditions create high frequency fatigue cycling superimposed over the mean diurnal change in strain.

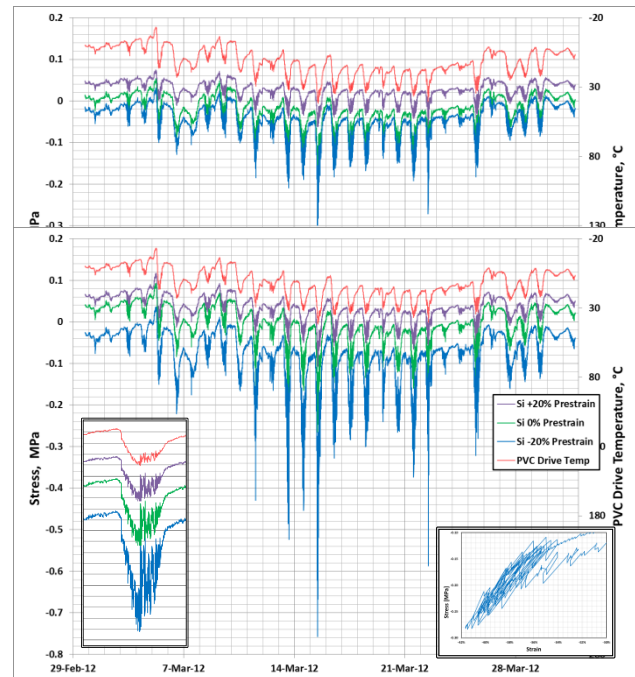


Figure 3: Month of daily cycles for polyurethane sealant (top) and silicone sealant (bottom). PVC drive temperature is shown in red. Insets in the bottom left hand and right hand side of the figure is the stress versus time and stress versus strain responses, respectively, of the silicone sealants during March 17, 2012.

Our prior research highlighted the ability of these test instruments to detect and track the buildup of compression set in situ.² This is experienced as an increasing tensile

stress at zero strain. Compression or tension set (CTS) is the result of stress relaxation of the sealant during prolonged exposure to hot or cold temperatures, respectively. It is detrimental to the sealant's ability to withstand rapid temperature reversal due to a magnification of the induced strain. The CTS values were recorded at zero strain just before the tensile stress relaxation period from which the tensile E_a (E_{aT}) value is calculated. The CTS for polyurethane and silicone sealants exposed to +20, 0, and -20% prestrains on IIIb are shown in the top and bottom of figure 4, respectively, along with the PVC drive temperature. The urethane sealant's stress at zero strain responds significantly more to temperature fluctuations than the silicone sealant. Both sealants exhibited higher CTS during the summer. The +20% prestrain specimens impart tensile stresses at zero strain. This is indicative of the prestrain not being entirely nullified by stress relaxation during the test period. Similar to all specimens this stress decreased during the winter. The 0% prestrain specimens started with near zero CTS and trended towards increasing compression CTS values in the summer with some recovery during the winter. The -20% prestrain samples started at high compressive stresses at zero strain with full relaxation taking place within 10 months. Thus, the -20% prestrain appears to have succumbed to stress relaxation with further changes in CTS mirroring the 0% preset samples.

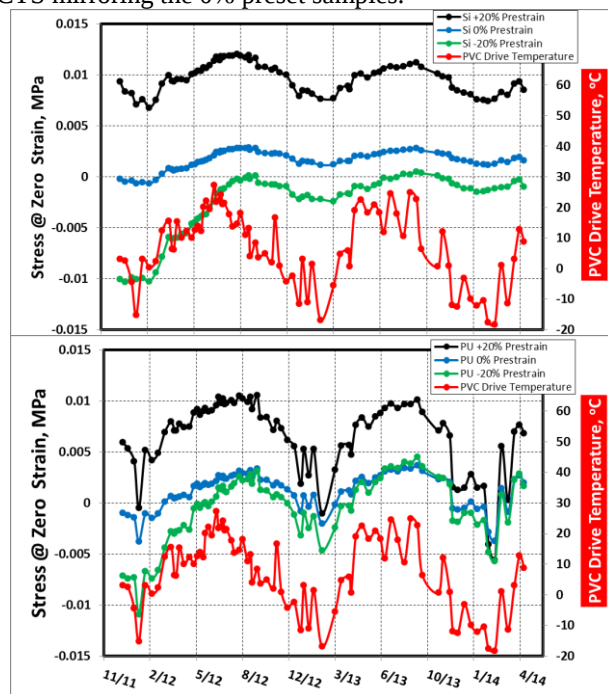


Figure 4: CTS and PVC drive temperature (right hand axis) versus exposure time for silicone (top) and urethane (bottom) sealants from IIIb.

The E_a values for all of the sealants specimens are plotted in figures 5 through 10. Each of these figures includes E_{aT} and compression E_a (E_{aC}) values for all replicates during the aging period with the initial ambient temperature at the E_a cycle start. The data from specimens run on IIIa is not included after LVDT failure in January of 2013. As noted in the preceding discussion the urethane

sealants exhibit significantly more response to changes in temperature than the silicone sealants. Dynamic mechanical analysis of sealant thin films reveals a downward sloping rubbery plateau for the urethanes where the silicone has an approximately flat modulus response. The silicone sealants underwent increases in compliance, figures 5-7, evident as a change in E_{aT} and E_{aC} to lower absolute values during early stages of aging followed by more consistent behavior. These E_a values were calculated at the actual strains that include the prestrains even though the instruments imposed a uniform $\pm 10\%$ strain to all specimens during stress relaxation.

The inclusion of compressive stress relaxation allows direct comparison with the E_{aT} values for the zero prestrain samples, figures 6 and 9. Deviations from symmetry should allow detection of delamination or large crack formation. Indeed this did facilitate the detection of the failed specimens on IIIa after LVDT failure. The remaining specimens on IIIb maintain good symmetry through the testing period indicating no gross delamination or crack formation. The $\pm 20\%$ prestrain samples exhibit asymmetry between the E_{aC} and E_{aT} values. The +20% prestrain specimens have greater compressive E_a values than the tensile and vice versa for the -20% prestrain specimens indicative of tensile and compressive set, respectively.

Conclusions

Automated outdoor strain cycling methodology allowed tracking of daily strain cycles and the superimposed erratic strain fatigue induced by rapid changes in radiative heat flux. Frequent measurement of zero strain loads provide a continuous record of changes in CTS. The silicone sealant underwent significant compliance increase during aging while the urethane maintained its modulus. The urethane sealant responded significantly to temperature changes in accordance with its sloping rubbery plateau behavior. E_{aT}/E_{aC} comparisons allowed detection of delamination after LVDT failure. This results of this research indicate that installation of building sealants such that a negative strain is present, e.g. winter application, acts to amplify hot compression strains and loads.

Acknowledgements

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References

1. ASTM C719-14, Standard Test Method for Adhesion and Cohesion of Elastomeric Joint Sealants Under Cyclic Movement (Hockman Cycle). ASTM International: West Conshohocken, PA, 2014.
2. Schueneman, G.; Hunt, C.; Lacher, S.; White, C.; Hunston, D., In Situ Measurement of Compression Set in Building Sealants During Outdoor Aging. *Journal of ASTM International* 2011, 9 (1), 11.
3. Treloar, L. R. G., *Physics of Rubber Elasticity*. 3rd ed.; Oxford University Press: Oxford, UK, 2002; Ferry, J. D., *Viscoelastic Properties of Polymers*. 3rd ed.; John Wiley & Sons, Inc: New York, NY, 1980.

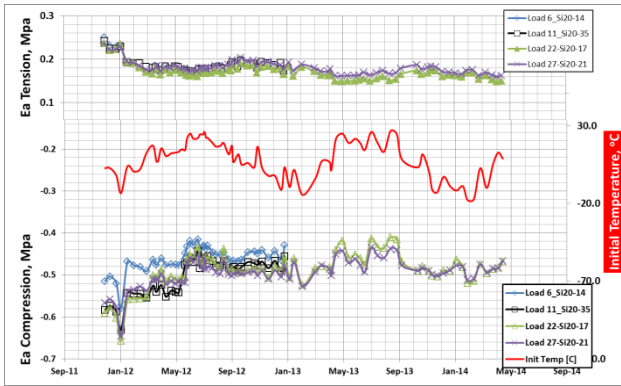


Figure 5: Apparent modulus of silicone sealants with +20% preset strain during aging measured in tension (top) and compression (bottom), and ambient temperature at start of the test.

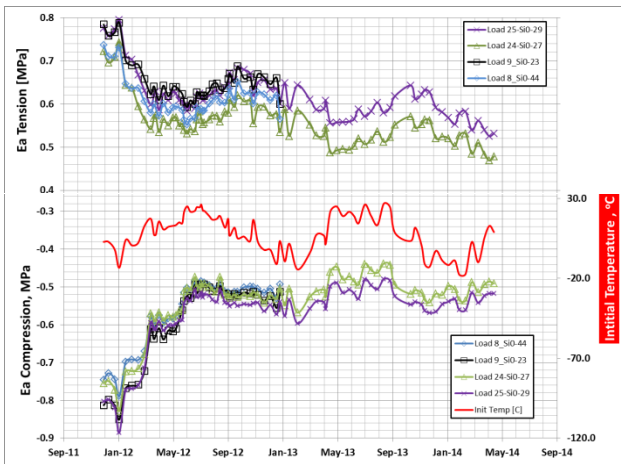


Figure 6: Apparent modulus of silicone sealants with zero preset strain during aging measured in tension (top) and compression (bottom), and ambient temperature at start of the test.

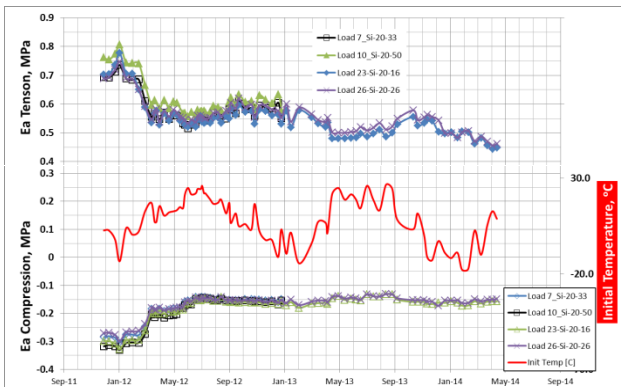


Figure 7: Apparent modulus of silicone sealants with -20% preset strain during aging measured in tension (top) and compression (bottom), and ambient temperature at start of the test.

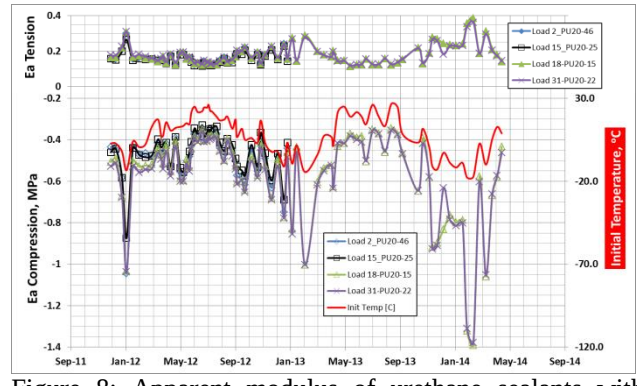


Figure 8: Apparent modulus of urethane sealants with +20% preset strain during aging measured in tension (top) and compression (bottom), and ambient temperature at start of the test.

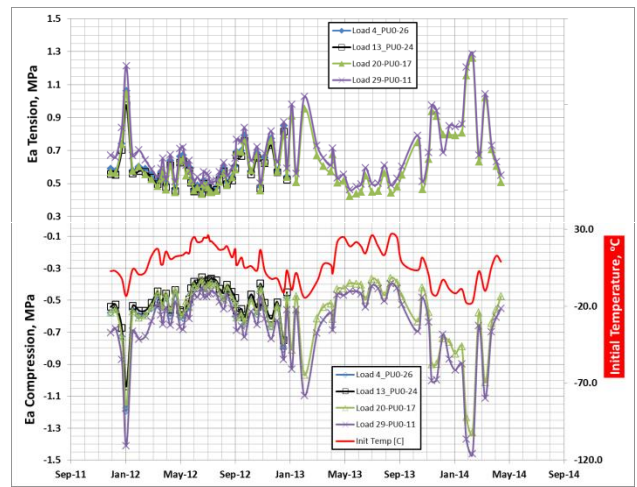


Figure 9: Apparent modulus of urethane sealants with zero preset strain during aging measured in tension (top) and compression (bottom), and ambient temperature at start of the test.

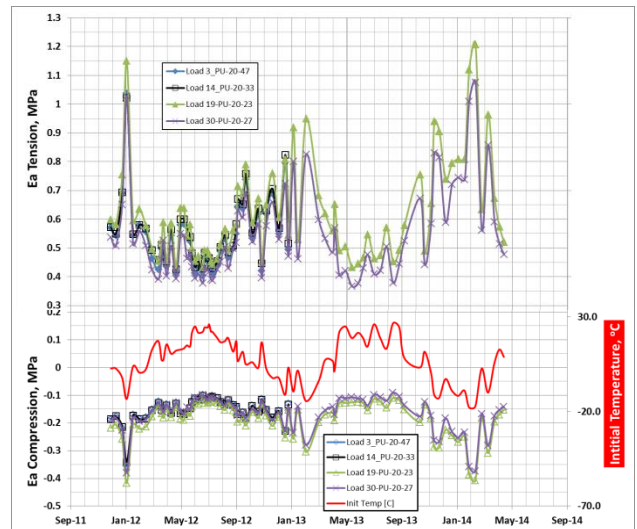


Figure 10: Apparent modulus of urethane sealants with -20% preset strain during aging measured in tension (top) and compression (bottom), and ambient temperature at start of the test.